

## EXHIBIT A

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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: Application of LEE et al.

Application No.: 10/716,369

Examiner: Moore, Margaret G.

Date Filed: November 18, 2003

Group: 1712

For: PROCESS FOR SIBCN BASED PRECERAMIC POLYMERS  
AND PRODUCTS DERIVABLE THEREFROM

## CERTIFICATE UNDER 37 CFR 1.8(a)

I hereby certify that this correspondence addressed to Mail Stop  
RCE, Commissioner for Patents is being transmitted via facsimile  
No. 703-872-9406 on 1/5/06

Neil R. Jeuer

Reg No 46,803

RULE 132 DECLARATIONMS RCE  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

I, Gary E. LeGrow, declare as follows:

1. I am familiar with the above-referenced patent application No. 10/716,369 entitled "PROCESS FOR SIBCN BASED PRECERAMIC POLYMERS AND PRODUCTS DERIVABLE THEREFROM" (hereafter the '369 application), and the subject matter described therein, as well as the provisional application filed in this case.

2. As detailed in the attached cv, I have substantial expertise in synthesis of preceramic polymers. For example, a monograph entitled "Fiber Reinforced Ceramic Composites" edited by K.S. Mazdiyarni and published by Noyes Publications in 1990. ISBN 0-8155-1333-3 is relevant in this regard. Chapter 2 of this monograph is authored by N.R. Langley, G.E. LeGrow (myself) and J. Lipowitz entitled "Properties of ceramic fibers from Organosilicon

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Polymers" has a specific reference to chemistry relevant to the '369 application on Page 66 (middle of the page – equations 11 and 12).

3. I have been asked to provide an opinion whether when the '369 application teaches one having ordinary skill in the art processes for synthesizing pre-ceramic polymers having a residual chlorine content of 100 ppm or less, as synthesized. The "as synthesized" aspect refers to a process which removes the need for distillation or filtration to remove the Cl which is characteristic of known related processes.

4. The chemical method of removal of chlorine, the chlorine being originally attached to either Boron or Silicon in  $\text{BCl}_3$  or  $\text{HSiCl}_3$  respectively, in the synthesis method according to the '369 application is clearly taught in Figure 4(a)-(e) of the provisional application, as well as beginning in paragraph 43 of the published application, specifically the volatilization of  $\text{Me}_3\text{SiCl}$  at  $58^\circ\text{C}$ . Since the reaction is conducted over a range of  $125 - 300^\circ\text{C}$  with  $\text{Me}_3\text{SiCl}$  which has a boiling point of  $58^\circ\text{C}$ , Cl should only be present in the product in trace quantities at the end of the reaction.

5. Moreover, use of a stoichiometric excess of HMDZ is taught in the disclosure in paragraph 31 of the published application. HMDZ is the source of  $\text{Me}_3\text{Si-}$  groups necessary for the formation of  $\text{Me}_3\text{SiCl}$ . HMDZ is also the source of  $-\text{NH-}$  components in the final preceramic polymer.

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6. In addition, at higher temperatures during the synthesis process, ie: above 127°C, any residual excess HMDZ will be volatilized and removed from the reactor. This is also taught in paragraph 43 of the published application.

7. Another relevant point is that most of the processes described in the related art utilizing  $\text{HSiCl}_3$  or other chlorosilanes and HMDZ to form preceramic polymers do not use solvents. In contrast, the '369 application teaches use of hexane as a solvent to disperse the developing preceramic polymer. I believe that the use of hexane (or other suitable solvent) aids in the efficient removal of  $\text{Me}_3\text{SiCl}$  from the system.

8. An aspect regarding the avoidance of formation of  $\text{NH}_4\text{Cl}$  in pre-ceramic polymer synthesis is the exclusion of water. The procedures taught for the exclusion of water in the synthesis of this preceramic polymer according to the invention are rigorously disclosed beginning in paragraph 75 where Schlenk-type glassware is described.

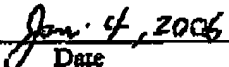
9. In conclusion, for the reasons noted above, it is my opinion that the '369 application clearly teaches one having ordinary skill in the art processes for synthesizing pre-ceramic polymers having a residual chlorine content of 100 ppm or less, as synthesized without the need for further processing to remove Cl.

10. I have no financial interest in the present invention and am not being compensated for rendering this Declaration.

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11. I further state that all statements made herein are of my own knowledge and true and that all statements made on information and belief are believed to be true; and further that these statements were made with my knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

  
Dr. Gary E. LeGrow

  
Date

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## Gary Edward LeGrow (GEL)

### Degrees:

B.A. (Mathematics, Physics and Chemistry), University of Toronto (Canada), 1960  
M.A. (Synthetic Organosilicon Chemistry), University of Toronto (Canada), 1962  
Ph.D. (Physical Organosilicon Chemistry, Kinetics) University of Toronto (Canada) 1964

All three degrees were obtained under the guidance of Prof. Adrian G. Brook, a recipient of the prestigious ACS Fredrick Stanley Kipping Award in Organosilicon Chemistry.

**Link to the Chemistry Dept. of University of Toronto, Canada:** [www.chem.u.toronto.ca](http://www.chem.u.toronto.ca)

### Biographical Information:

1964-1965: Dr. LeGrow held a Post Doctorate position at the University of Sussex (England) with Prof. Colin Eaborn, studying the synthesis and thermal decomposition of metallorganosiloxanes. Prof. Eaborn was the author of the first textbook on Organosilicon Chemistry and was also a recipient of the ACS Fredrick Stanley Kipping Award in Organosilicon Chemistry.

**Link to the Chemistry Dept. of the University of Sussex, England:**  
[www.cpes.sussex.ac.uk/chemistry/index.html](http://www.cpes.sussex.ac.uk/chemistry/index.html)

1965-1995: Dr. LeGrow held numerous positions in the Research and Technical Service and Development Departments of Dow Corning Corporation in Midland, MI and Carrollton, KY. His first position as Research Chemist was under the guidance of Dr. John L. Speier, the only industrial chemist to ever receive the ACS Fredrick Stanley Kipping Award in Organosilicon Chemistry. The early part of his career (1965-1970) was primarily directed at synthetic organofunctional silicon chemistry. From 1970-1980, his research was primarily directed at the synthesis of novel organosilicone resins, their characterization and their use in industrial and military applications. From 1980-1984, his research was directed at the fundamental science of the most basic reaction in the field of organosilicone chemistry, the Direct Process. From 1984-1989, as a Research Scientist, his research was directed towards pre-ceramic polymers and their conversion to materials of construction of importance to the military and aerospace industries. In 1989, he joined the Technical Service and Development Department, and as a Development Scientist led a global development program on new organosilicone materials for the cosmetic industry. From 1994-1995, he continued as a Development Scientist in the Advanced Materials Section of the Business Research Department. After 30+ years, Dr. LeGrow retired from Dow Corning

at the end of 1995.

**Link to Dow Corning Corporation:** [www.dowcorning.com](http://www.dowcorning.com)

1996-2002: Dr. LeGrow accepted the position of Principal Scientist in the Research and Development Department of PCR, Inc., Gainesville, FL, a company with 47 years of research, development and manufacturing history in the field of Organosilicon Chemistry. PCR, Inc. is now part of the largest specialty chemical company in the world - Clariant. Dr. LeGrow led a research group, primarily directed at the creation and application development of novel organosilicone materials. Dr. LeGrow retired Dec. 31, 2003.

**Link to Clariant:** [www.clariant.com](http://www.clariant.com) or [www.na.clariant.com](http://www.na.clariant.com)

2002-2003 Dr. LeGrow accepted the faculty position of Courtesy Scientist in the Materials Science and Engineering (MSE) Department of the University of Florida (UF), effective Nov. 1, 2002. Dr. LeGrow briefly conducted a materials Research and Development program utilizing the unique properties and benefits of organosilicone technology. Dr. LeGrow retired from this position on Nov. 1, 2003.

**Link to the MSE Dept. at UF:** [www.mse.ufl.edu](http://www.mse.ufl.edu)

During his career Dr. LeGrow published 25 scientific papers, contributed to several monographs, and is an inventor of 71 US patents and numerous foreign patents.

#### Publications

#### Patents

During his career Dr. LeGrow was also an adjunct professor at Michigan State University, Saginaw Valley State University and Delta College for several years. During this time he taught courses in Advanced Organic Chemistry, and numerous courses in Organosilicon Chemistry. He also taught a course in Designed Experimentation for Dow Corning over a period of almost 20 years in the USA, Europe, Japan, Australia and China.

Dr. LeGrow is a 45 year member of the American Chemical Society, a member of the Society of Cosmetic Chemists and a member of the International Nomenclature Committee of the Cosmetic, Toiletries and Fragrances Association.

**Link to the American Chemical Society:** [www.acs.org](http://www.acs.org)

**Link to the Society of Cosmetic Chemists:** [www.sconline.org](http://www.sconline.org)

**Link to the Cosmetics, Toiletries and Fragrances Association:** [www.ctfa.org](http://www.ctfa.org)

**Link to the International Federation of Society of Cosmetic Chemists: [www.ifsc.org](http://www.ifsc.org)**

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## Gary E. LeGrow, Ph.D.

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11. LeGrow, G.E., Lipowitz, J., STRUCTURE AND PROPERTIES OF SILICON CARBIDE/NITRIDE CERAMIC FIBERS FROM POLYMER PRECURSORS, The Fiber Society, Ashville, NC, Spring (1988)
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15. LeGrow, G.E., Glover, D.A., Madore, L.M., Sojka, M., Taylor, R.B., WATER SOLUBLE DIMETHICONE COPOLYOL WAXES FOR THE PERSONAL CARE INDUSTRY, PROCEEDINGS of the 17th IFSCC Congress, Yokohama, Japan (1992)
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**Gary E. LeGrow, Ph.D.**  
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